

## Claims

1. A process for making rare earth doped optical fibre using stable dispersions (sol) of RE coated silica nanoparticles and applying a thin coating of the said silica sol containing suitable dopants selected from Ge, Al, P etc.
2. A process for making rare earth doped optical fibre, said process comprising steps of:
  - (a) obtaining rare earth oxide doped silica nanoparticles by sonochemical method;
  - (b) preparing stable dispersions of the above RE containing powders in the desired proportion in presence of suitable dopants like  $\text{Al}^{3+}$ ,  $\text{Ge}^{4+}$  etc. in a silica sol of  $\text{Si}(\text{OC}_2\text{H}_5)_4$  under sonication.;
  - (c) applying a thin coating of silica sol on the inner surface of high purity clear fused silica glass tubes by sol-gel dip coating technique;
  - (d) drying the coated layer in air at  $70^\circ$  to  $150^\circ\text{C}$ ;
  - (e) mounting the tube on glass working lathe for processing by MCVD technique;
  - (f) dehydrating the coated layer inside the tube at a temperature in the range of  $800$ - $1200^\circ\text{C}$  in presence of excess  $\text{Cl}_2$ ;
  - (g) sintering the coated layer in presence of a mixture of oxygen and helium in the temperature range of  $1400$  to  $1750^\circ\text{C}$  for formation of the core;
  - (h) heating the tube gradually up to a temperature of  $1900^\circ\text{C}$  for further consolidation of the sintered layer;
  - (i) collapsing the tube by usual method at a temperature in the range of  $2000$ - $2300^\circ\text{C}$  to obtain a preform;
  - (j) overcladding the preform with silica tube, and
  - (k) drawing fibres of standard dimensions from the preform by the conventional methods.
3. A process as claimed in claim 1 wherein, the RE oxide is selected from  $\text{Eu}_2\text{O}_3$ ,  $\text{Nd}_2\text{O}_3$ ,  $\text{Tb}_2\text{O}_3$  and  $\text{Er}_2\text{O}_3$  for preparation of the silica nanoparticles.
4. A process as claimed in claim 1 wherein,  $\text{P}_2\text{O}_5$  and F doped synthetic cladding is deposited within a silica glass substrate tube prior to development of the coating by

known method like Modified Chemical Vapour Deposition (MCVD) process to obtain matched or depressed clad type structure in the preform.

5. A process as claimed in claim 1 wherein, the particle size of the RE coated  $\text{SiO}_2$  powders ranges from 50 to 200 nm.
6. A process as claimed in claim 1 wherein, the composition in oxide mol% of  $\text{SiO}_2$ :  $\text{Re}_2\text{O}_3$  in  $\text{Re}_2\text{O}_3$  coated  $\text{SiO}_2$  powders varies from 99.5 : 0.5 to 95 : 5.
7. A process as claimed in claim 1 wherein, the equivalent oxide mol% of  $\text{SiO}_2$  in the dispersion varies from 98.5 to 90.5.
8. A process as claimed in claim 1 wherein, a silica sol prepared with  $\text{Si}(\text{OC}_2\text{H}_5)_4$  was used as the diluent of the  $\text{RE}_2\text{O}_3$  coated silica powder.
9. A process as claimed in claim 1 wherein, the equivalent oxide mol% of  $\text{GeO}_2$  in the dispersion varies from 1.0 to 5.0 .
10. A process as claimed in claim 1 wherein,  $\text{Ge}^{4+}$  was added through  $\text{Ge}(\text{OC}_2\text{H}_5)_4$  in the silica sol.
11. A process as claimed in claim 1 wherein, the equivalent oxide mol% of  $\text{Al}_2\text{O}_3$  in the dispersion ranges from 0.5 to 4.0.
12. A process as claimed in claim 1 wherein,  $\text{Al}_2\text{O}_3$  is provided to the solvent in the form of aluminium salts such as chlorides, nitrates or any other salt soluble in the solvent.
13. A process as claimed in claim 1 wherein, the solution of aluminium salt is prepared using a solvent selected from alcohol and water.
14. A process as claimed in claim 1 wherein, the oxide mol% of  $\text{Er}_2\text{O}_3$  in the dispersion ranges from 0.01 to 0.60.
15. A process as claimed in claim 1 wherein, strong mineral acids used for preparing the sol for dispersion are selected from hydrochloric or nitric acid.
16. A process as claimed in claim 1 wherein, the alcohol selected is soluable in the dispersion system.
17. A process as claimed in claim 1 wherein, the alcohol is selected from the group comprising of methyl alcohol, ethyl alcohol, propan-1-ol, propan-2-ol, butan-1-ol and butan-2-ol.

18. A process as claimed in claim 1 wherein, pH of the dispersion ranges from 1 to 5.
19. A process as claimed in claim 1 wherein, viscosity of the dispersion varies from 1 to 10 mPa s.
20. A process as claimed in claim 1 wherein, sonication time of the dispersion ranges from 30 to 200 minutes.
21. A process as claimed in claim 1 wherein, settling time of the dispersion varies from 1 to 10 hours.
22. A process as claimed in claim 1 wherein, lifting speed of the tube from the dispersion ranges from 4 to 15 cm/minutes.
23. A process as claimed in claim 1 wherein, baking temperature of the coated tube varies from 70° to 150°C.
24. A process as claimed in claim 1 wherein, baking time of the coated tube ranges from 0.5 to 5 hours.
25. A process as claimed in claim 1 wherein, the core composition is selected from the group comprising of  $\text{RE}_2\text{O}_3 + \text{SiO}_2 + \text{GeO}_2$ ,  $\text{RE}_2\text{O}_3 + \text{SiO}_2 + \text{GeO}_2 + \text{Al}_2\text{O}_3$ ,  $\text{RE}_2\text{O}_3 + \text{SiO}_2 + \text{GeO}_2 + \text{Al}_2\text{O}_3 + \text{P}_2\text{O}_5$  and  $\text{RE}_2\text{O}_3 + \text{SiO}_2 + \text{GeO}_2 + \text{P}_2\text{O}_5$ .
26. A process as claimed in claim 1 wherein, the temperature of the RE oxide containing core layer is increased in steps of 50 to 200°C during sintering depending on the composition and Al/RE concentration of the core layer.
27. A process as claimed in claim 1 wherein, the mixture of  $\text{O}_2$  and He is in the range of 3:1 to 9:1 during sintering.
28. A process as claimed in claim 1 wherein, source of chlorine is  $\text{CCl}_4$  where helium is used as carrier gas.
29. A process as claimed in claim 1 wherein, the proportion of  $\text{Cl}_2$ :  $\text{O}_2$  during drying varies from 1.5 : 1 to 3.5 : 1.
30. A process as claimed in claim 1 wherein, the dehydration period lies between 1 to 2 hours.
31. A process as claimed in claim 1 wherein, the core layer is sintered in the presence of germania to facilitate germania incorporation and to obtain appropriate numerical aperture value.

32. A process as claimed in claim 1 wherein, germania is supplied to the core layer during sintering by including  $\text{GeCl}_4$  with the input oxygen.
33. A process as claimed in claim 1 wherein, the sintering is carried out at a temperature of  $1200^\circ\text{C}$  to  $1400^\circ\text{C}$ .
34. A process as claimed in claim 1 wherein, depending on the composition of the core,  $\text{POCl}_3$  is added to the input gas mixture during sintering.
35. A process as claimed in claim 1 wherein, the core layer is doped with  $\text{P}_2\text{O}_5$  to facilitate RE incorporation.
36. A process as claimed in claim 1 wherein,  $\text{P}_2\text{O}_5$  and  $\text{GeO}_2$  concentrations vary from 0.5 to 5.0 mol% and 3.0 to 25.0 mol% respectively in the RE doped core layer.
37. A process as claimed in claim 1 wherein, the numerical aperture of the fibre is varied from 0.10 to 0.30.
38. A process as claimed in claim 1 wherein, RE concentration in the core is maintained in the range of 50 to 4000 ppm to produce fibres suitable for application as amplifiers, fibre lasers and sensors or different purposes.
39. A process as claimed in claim 1 wherein, codopants like Al and other rare earths are added to the core doped with a selected RE to fabricate fibres containing various dopants in the core in the concentration range 50 to 5000 ppm and numerical aperture varying between 0.10 and 0.30.
40. A process as claimed in claim 1 wherein, the deposition of a porous soot layer at high temperature ( $1000^\circ\text{C}$  or above) by CVD process inside a fused silica glass tube or on a seed rod (VAD or OVD apparatus) is eliminated for formation of the core.
41. A process as claimed in claim 1 wherein, better control is achieved over the characteristics of the coated layer like porosity, thickness etc. and uniformity along the length of the tube.
42. A process as claimed in claim 1 wherein, the difficulties and uncertainties involved in incorporation of the rare-earth ions in desired concentration into the porous soot layer by the solution-doping technique and such other methods are eliminated.

43. A process as claimed in claim 1 wherein, the rare-earth oxide coated silica nanoparticles are dispersed at ambient temperature in the silica sol mentioned above under sonication thereby eliminating the possibility of formation of the microcrystallites and clusters of rare-earth ions as in the conventional techniques.
44. A process as claimed in claim 1 wherein, the possibility of evaporation of RE salts at high temperatures is considerably eliminated due to the direct addition of RE oxides which prevents change in composition including variation of RE concentration in the core and also reduces the possibility of formation of RE dip at the core centre.
45. A process as claimed in claim 1 wherein, the process ensures better control of RE concentration in the doped region and homogeneous distribution of RE ions along the radial direction as well as throughout the length of the preform.
46. A process as claimed in claim 1 wherein, the RE incorporation efficiency is much higher compared to the conventional techniques because of direct addition of the RE oxides into the dispersion instead of the corresponding salt by the conventional techniques thereby minimising the possibility of evaporation and change in concentration.
47. A process as claimed in claim 1 wherein, the addition of  $\text{Ge}(\text{OET})_4$  at ambient temperature in the silica sol above reduces the quantity of  $\text{GeCl}_4$  which is required at high temperature to achieve the desired NA.
48. A process as claimed in claim 1 wherein, the time period of processing the silica tube at high temperature and the number of steps involved for doping of RE ions by the conventional techniques for fabrication of the preform are considerably reduced.
49. A process as claimed in claim 1 wherein, the processing of the tube at ambient temperature before sintering and collapsing instead of high temperature involved in the CVD process makes the process less sensitive to the process parameters unlike the conventional processes.
50. A process as claimed in claim 1 wherein, the advantages described above increases the reproducibility and reliability of the process to a great extent.

51. A process as claimed in claim 1 wherein, the requirement of precision equipments for control of porous soot deposition, RE incorporation etc. during fabrication of the preform is considerably eliminated which will reduce the capital investment and cost of the product.
52. A process as claimed in claim 1 wherein, the advantages combined make the process simple and more economic than the conventional processes.

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